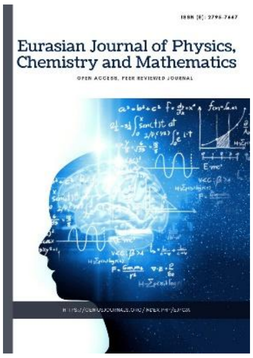

Effect of Residual Carbon on Vanadium and Scandium Leaching from Thermally and Steam-Thermally Treated Metalliferous Oil Shale

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ABSTRACT

The leachability of vanadium and scandium was compared for two technologically distinct products derived from metalliferous oil shale of the Kyzylkum region, Uzbekistan: thermal-treatment ash produced without a dedicated decarbonization step and flotation tailings from steam-thermally treated shale after removal of the carbonaceous fraction. Two existing sulfuric-acid leaching datasets were evaluated under closely comparable conditions. At a liquid-to-solid ratio of 3 mL g^{-1}, 75–80 °C, and approximately 60 min, average vanadium recovery was 35.8% from thermal ash and 74.7% from flotation tailings, whereas scandium recovery was 31.5% and 78.8%, respectively. Maximum recoveries within the investigated parameter ranges were 56.7% versus 90.0% for V and 54.8% versus 98.6% for Sc. The thermal ash retained residual organic matter, reflected by a loss on ignition of 11.48%, while the flotation product had undergone targeted decarbonization. The markedly lower recovery from the carbon-bearing ash is therefore consistent with parasitic re-sorption of dissolved metal species by residual carbon, a mechanism previously observed for related material. For the decarbonized tailings, a 4×4 factorial dataset yielded statistically significant effects of both sulfuric-acid concentration and leaching time: R^2 = 0.864 for V and 0.965 for Sc, with $p < 0.05$ for both factors. The results identify decarbonization as a quantitatively important process variable and support its direct experimental evaluation as a pretreatment step for improving V and Sc recovery from thermally treated oil-shale residues.

Keywords: oil shale; residual carbon; decarbonization; sulfuric acid leaching; vanadium; scandium

Highlights

- Decarbonized flotation tailings show markedly higher V and Sc leachability.
- Average V recovery increases from 35.8% in ash to 74.7% in tailings.
- Average Sc recovery increases from 31.5% in ash to 78.8% in tailings.
- Residual carbon in thermal ash is consistent with parasitic metal re-sorption.
- A balanced factorial design resolves acid-concentration and time effects.

1. Introduction

Pretreatment history can strongly influence the subsequent hydrometallurgical behavior of carbonaceous mineral feeds. Thermal processing may remove part of the organic matter and transform metal-bearing mineral phases, but the extent of decarbonization depends on the process conditions and may remain incomplete. Residual carbon is particularly important when the target metals are present at low concentrations because carbonaceous matter can alter reagent consumption, wetting, redox conditions, and the apparent distribution of dissolved species between the liquid and solid phases.

Metalliferous oil shales of the Kyzylkum basin in Uzbekistan have attracted interest as a potential source of uranium, vanadium, scandium, rhenium, and other metals. Reported oil-shale resources in Uzbekistan are on the order of 47 billion tonnes [1,2]. Several processing routes have been proposed for this feed class, including steam-thermal treatment followed by flotation and hydrometallurgical processing [3], as well as thermal activation of refractory black-shale ores followed by sulfuric-acid leaching [4]. However, direct quantitative comparisons between differently pretreated products remain limited.

A second methodological issue is the design of leaching experiments. Balanced factorial designs permit the independent contribution of operating variables to be separated statistically, whereas incomplete or unbalanced datasets can obscure otherwise real effects. Modern mineral-leaching studies increasingly combine design of experiments, regression, response-surface analysis, and data-driven modeling to improve process interpretation and optimization [5,6].

This study compares vanadium and scandium leachability in two products of related Kyzylkum oil-shale processing: (i) thermal-treatment ash with residual organic matter and no dedicated decarbonization step, and (ii) flotation tailings from steam-thermally treated shale after removal of the carbonaceous fraction. The objectives were to quantify the difference in recovery under comparable leaching conditions, assess the consistency of the observed behavior with a residual-carbon re-sorption mechanism, and examine how experimental-design quality affects the statistical resolution of sulfuric-acid concentration and leaching-time effects.

2. Materials and methods

2.1 Compared materials and datasets

The analysis combined two sulfuric-acid leaching datasets reported for related metalliferous oil-shale products from the Kyzylkum region. Dataset A comprised 28 tests on thermal-treatment ash. The investigated ranges were 10–100 g L^{-1} H_2SO_4, 30–75 °C, 1–2 h, and liquid-to-solid ratios of 3–5 mL g^{-1}. Dataset B comprised 16 tests on flotation tailings from steam-thermally treated shale, arranged as a full 4×4 factorial design with H_2SO_4 concentrations of 50–200 g L^{-1} and leaching times of 30–120 min at 80 °C, using 50 g charges and L/S = 3 mL g^{-1} [3].

For direct comparison, observations were selected from both datasets at closely matched conditions: L/S = 3 mL g^{-1}, temperature 75–80 °C, and leaching time close to 60 min. The thermal-treatment ash retained residual organic matter, with a reported loss on ignition of 11.48% compared with 30.26% for the original shale. The flotation-tailings route included a targeted removal of the carbonaceous fraction.

2.2 Response variables and statistical analysis

Vanadium and scandium recovery to the aqueous phase were used as the response variables. For the flotation-tailings dataset (n = 16), linear regression models were reported as $V = 44.26 + 0.061C + 0.295\tau$ and $Sc = 50.13 + 0.115C + 0.239\tau$, where C is the H_2SO_4 concentration in g L^{-1} and τ is the leaching time in minutes. Model quality was assessed using R^2 and factor p-values. The thermal-ash dataset was not a complete factorial design; the associated analysis reported non-significant duration effects with p-values ranging from 0.53 to 0.99.

Because the two datasets were generated for different pretreatment routes and were not a paired experiment designed specifically to isolate residual carbon as the only changing variable, the comparison is interpreted as mechanistic evidence rather than definitive causal proof. A controlled decarbonization experiment on the same ash would be required to quantify the carbon effect independently of other pretreatment-induced mineralogical changes.

3. Results and discussion

3.1 Quantitative comparison of V and Sc leachability

Under the selected comparable conditions, the flotation tailings consistently outperformed the thermal-treatment ash. Average V recovery increased from 35.8% for the ash to 74.7% for the flotation tailings, corresponding to approximately a twofold increase. The difference was even larger for Sc, for which average recovery increased from 31.5% to 78.8%, or roughly 2.5 times. The maxima within the investigated operating windows showed the same pattern: 56.7% versus 90.0% for V and 54.8% versus 98.6% for Sc.

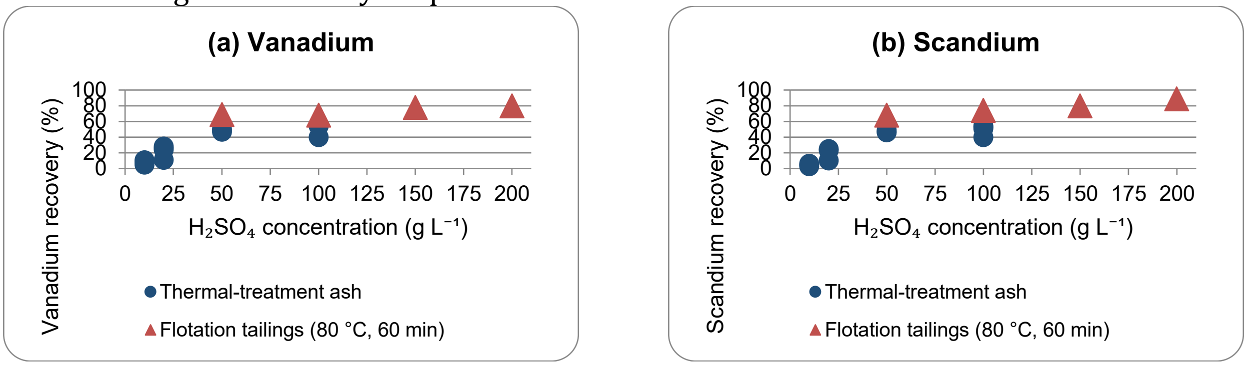

Figure 1. Comparison of vanadium (a) and scandium (b) recovery as a function of sulfuric-acid concentration for thermal-treatment ash and flotation tailings of steam-thermally treated shale. Chart objects and embedded data are editable in Word. Point-level values for the plotted comparison were reconstructed from the source figure because the manuscript did not provide a complete numerical table for every point; summary statistics are reported directly from the source dataset.

Table 1. Summary comparison of the two pretreated products under comparable leaching conditions.

Indicator	Thermal-treatment ash	Flotation tailings after steam-thermal treatment
Average V recovery, %	35.8	74.7
Average Sc recovery, %	31.5	78.8
Maximum V recovery, %	56.7	90.0
Maximum Sc recovery, %	54.8	98.6
Loss on ignition / carbon status	11.48% LOI; incompletely decarbonized	Targeted decarbonization; comparable LOI not reported
Experimental design	Incomplete	Full 4×4 factorial

3.2 Residual carbon and apparent metal loss

The thermal-treatment ash retained a measurable organic fraction, indicating that thermal processing did not completely remove carbonaceous matter. In contrast, the flotation-tailings route incorporated deliberate removal of the carbon-rich fraction. The source study reports an independent comparison on related material in which metal recovery remained below 40% without decarbonization but exceeded 90% after carbon removal [3]. This behavior is consistent with a parasitic or reverse-sorption mechanism in which metal ions that have already entered solution are re-captured by residual carbon and returned to the solid phase.

The recovery level observed for the thermal ash (approximately 32–36% on average for the two target elements) lies between the low-recovery non-decarbonized case and the >90% decarbonized case reported for the related material. The agreement is mechanistically plausible, but residual carbon should not be treated as the only possible cause. Thermal and steam-thermal pretreatments may also modify mineral phases, porosity, surface chemistry, and the accessibility of V- and Sc-bearing phases. These effects should be resolved in a purpose-designed paired experiment.

3.3 Influence of experimental-design quality on factor significance

The full 4×4 factorial dataset for the flotation tailings allowed the effects of acid concentration and leaching time to be separated statistically. For V, the regression model gave $R^2 = 0.864$, $p(C) = 0.011$, and $p(\tau) < 0.0001$. For Sc, $R^2 = 0.965$ and both factors were highly significant ($p < 0.0001$). Thus, within the investigated range, both acid concentration and time contributed measurably to recovery.

Table 2. Reported regression statistics for the flotation-tailings dataset (n = 16).

Element	Regression model	R^2	Factor significance
V	44.26 + 0.061C + 0.295τ	0.864	p(C)=0.011; p(τ)<0.0001
Sc	50.13 + 0.115C + 0.239τ	0.965	p(C)<0.0001; p(τ)<0.0001

For the incomplete thermal-ash design, the duration effect was not statistically resolved (reported $p = 0.53–0.99$). This contrast illustrates that the detectability of process effects depends not only on chemistry but also on the balance and coverage of the experimental plan. Future validation should therefore use a single coordinated factorial design in which decarbonization status, acid concentration, temperature, time, and L/S ratio are varied systematically.

3.4 Process implications and study limitations

The observed 2–2.5-fold difference in average recovery makes decarbonization a technically significant pretreatment candidate for thermally processed oil-shale residues. A practical route could involve flotation or another carbon-removal operation before acid leaching, provided that the additional separation cost is offset by increased metal recovery and reduced downstream losses. The principal limitation of the present comparison is that the two datasets were not generated as a common randomized experiment on identical material splits. Consequently, the estimated benefit should be regarded as a strong process-development hypothesis that now requires direct paired validation, carbon mass balance, mineralogical characterization before and after decarbonization, replicate leaching tests, and uncertainty reporting.

4. Conclusions

1. Under comparable sulfuric-acid leaching conditions, decarbonized flotation tailings gave approximately twice the average V recovery and 2.5 times the average Sc recovery obtained from incompletely decarbonized thermal-treatment ash.
2. The thermal ash retained residual organic matter (11.48% loss on ignition). The lower recovery is consistent with parasitic re-sorption of dissolved metals by residual carbon, although pretreatment-induced

mineralogical differences may also contribute.

3. For the full 4×4 flotation-tailings dataset, sulfuric-acid concentration and leaching time were statistically significant for both V and Sc, with R^2 values of 0.864 and 0.965, respectively.
4. The incomplete thermal-ash design did not statistically resolve the time effect, demonstrating the importance of balanced experimental design for mechanistic interpretation.
5. Decarbonization should be tested directly on thermal-treatment ash in a paired factorial experiment before the recovery gain is transferred to an industrial process design.

Declarations

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Data availability: The comparison is based on the experimental datasets cited in the manuscript; availability of the underlying point-level data should be specified by the author before submission.
Author contributions: S.K. Kurbanov — conceptualization, investigation, methodology, data analysis, interpretation, and manuscript preparation. [Please confirm or revise the CRediT roles before submission.]

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